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THE UNITED STATES PATENT OFFICE

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For : REFRACTORY SHAPED BODY WITH

INCREASED ALKALI RESISTANCE

Examiner : KARL E. GROUP

Commissioner for Patents

P. O. Box 1450

Alexandria, Virginia 22313-1450

Honorable Sir:

## **DECLARATION**

I, the undersigned, Dr. HANS-JURGEN KLISCHAT, am one of the inventors/applicants of the above U. S. Patent Application, and therefore, I am familiar with the present invention and the prior art thereof.

Furthermore, I, the undersigned, Dr. HANS-JURGEN KLISCHAT, believe I am a "person of knowledge", inasmuch as I am the Head of Research and Development at Refratechnik Cement GmbH, Germany, have a Doctor's Degree in Engineering in Germany, and I am an academically trained Mineralogist.

In that the U. S. Patent and Trademark Office Examiner, KARL E. GROUP, stated that the "experimental data set forth in the arguments is not persuasive in overcoming the rejection because it is not in declaration or affidavit form", this declaration setting forth the experimental data is set forth below.

All statements made of my own knowledge are true and all statements made upon information and belief are believed to be true, and further, these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent resulting therefrom.

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## Experiment concerning the microstructure (black core) of the refractory shaped body

High-alumina raw materials containing 51% by weight of  $Al_2O_3$  or 71% by weight of  $Al_2O_3$  were used to form refractory shaped bodies with different compositions. The maximum grain size was 4 mm. The grain size distribution corresponds to a typical fuller curve. These high alumina raw materials were mixed with 5% of a refractory bonding clay. The mixture also included 5% of SiC with the grain size from 0 to 0.9 mm. There were two refractory batches produced with a high-alumina raw material containing 51% by weight of  $Al_2O_3$  and two refractory batches with 71% by weight of  $Al_2O_3$ . These four mixtures were mixed with a binder component, whereby the two refractory batches with 71% by weight of  $Al_2O_3$  either contained 1.6% phosphoric acid and 1.2% water or 4% lignin sulfonate. The same components were mixed to the two refractory batches with 71% by weight of  $Al_2O_3$ . These four mixtures were pressed under a pressure of 90 MPa. Then, the shaped bodies were dried at a temperature of over 100 °C, and after subsequent drying the shaped bodies were fired at a sintering temperature of 1260 °C.

The pictures of ENCLOSURE I show the results after sintering, as follows:

PHOTO A: raw material with 51% Al<sub>2</sub>O<sub>3</sub> and lignin sulfonate;

PHOTO B: raw material with 71% Al<sub>2</sub>O<sub>3</sub> and lignin sulfonate;

PHOTO C: raw material with 71% Al<sub>2</sub>O<sub>3</sub> and phosphoric acid; and

PHOTO D: raw material with 51%  $Al_2O_3$  and phosphoric acid.

It is shown that the composition (PHOTO D) of the present invention has a much better microstructure than the other three compositions shown in PHOTOS A, B and C.

## Experiment concerning the alkali resistance

Crucibles were cut out of the four shaped bodies of the first experiment with an edge length of 70 mm and an internal bow with a diameter of 40 mm. To simulate alkali attack, these crucibles were filled with 70 g of potassium carbonate. The crucibles were then closed off with a cover made from the same material and were treated in a kiln for five hours at a temperature of  $1100^{\circ}$ C.

The results are shown in the pictures PHOTOS E, F, G and H of ENCLOSURE II.

The result after firing is that the microstructures of the comparative examples shown in PHOTOS E, F and G reveal infiltration. The shaped bodies were considerably destroyed by the alkali attack.

By contrast, the brick of the present invention shown in PHOTO H does not reveal any effect on the microstructure by the alkali attack. The shaped body is free of cracks. The alkali resistance of the shaped body according to the invention is, surprisingly, so high that potassium carbonate has boiled over out of the crucible, since it was unable to penetrate into the microstructure of the brick.

I declare that all statements made herein of my own knowledge are true; and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application for patent or document or patents resulting therefrom.

Dr. Hans-Jürger Klischat Göttingen, 2003-12-01